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GRAPHICAL TECHNIQUES FOR THE PRESENTATION OF INTERLABORATORY STUDY DATA - A DIAGNOSTIC AID

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ABSTRACT

Historically, most interlaboratory studies have documented current performance and evaluated the performance against a standard in tabular form. Graphical techniques of evaluation provide for a more immediate comparison of performance and a greater incentive for participants to improve their performance. Examples from a series of interlaboratory studies co-ordinated by Laboratory Services Branch of Environment Ontario during 1988-90 demonstrated this approach.

INTRODUCTION

The role of the interlaboratory or collaborative study to establish the precision of an analytical procedure is well established. The American Society for Testing and Materials (ASTM) and the Association of Official Analytical Chemists (AOAC) have published guidelines for conducting this type of study (1, 2). The data is treated and summarized using classical statistical techniques and an accepted standard of performance is established.

Interlaboratory performance studies are then used as a means of monitoring the ongoing performance of a group of analytical laboratories that are testing environmental samples. These studies have three functions: documentation of current performance, evaluation of that performance against a standard, and inducement to improve both individual and group laboratory performance

(3). There are many examples in the literature of interlaboratory performance studies that tabulate current performance and evaluate the performance against a standard using classical statistical techniques. Less frequently are the interlaboratory studies used to focus on ways to improve an individual laboratory's performance.

MOE LABORATORY SERVICES BRANCH APPROACH

The Laboratory Services Branch (LSB) of Environment Ontario initiated a series of Interlaboratory Performance Management Studies in 1988 in support of the ministry's Municipal and Industrial Strategy for Abatement (MISA) Program. The studies were designed not only to document the current level of performance of laboratories capable of performing MISA analyses, but also to be used as a tool for improving an individual laboratory's performance. Each study focused on a specific MISA analytical test group or groups (4), with different tests being repeated over time so as to monitor improvements in performance.

The traditional approach of reporting interlaboratory means, medians, and standard deviations for the various parameters has been included for the LSB studies, as they provide a familiar form of comparison for the participants. However the assessment of each individual laboratory's performance, and the recommendations for improvement are based on different graphical techniques.

The Youden two-sample plot (2) is a well-established technique for assessing intralaboratory precision. Traditionally it has been applied to single-parameter tests. The standard deviation of the results can be used to calculate a limit for acceptable performance, with outlying laboratories flagged (Figure 1). This approach works effectively for most conventional and inorganic analyses.

By applying the Youden two sample technique to the results from an organic gas chromatographic (GC) scan, the interanalyte repeatability and bias may be assessed for an individual laboratory (Figure 3). Tolerance limits may be assigned or calculated, to rank the precision and the bias of the data set. Outlying parameters may be flagged, as opposed to flagging an outlying laboratory.

To assess a laboratory's performance across a GC scan, the results from a sample may be converted to percent recovery of the design value, arranged in elution order, and presented as a bar graph (Figure 5). Trends across the scan may be noted, with recommendations for improvement. This may include patterns of increasing or decreasing recovery, suggesting adjustments for the GC temperature programming. A specific group of parameters within the scan may be over- or under-recovered, suggesting problems with extraction efficiency. Individual problem parameters are easily identified, and steps can be taken to check calibration standards or the

appropriateness of the method for a specific parameter.

The following examples from several different LSB Interlaboratory Studies demonstrate how these graphical techniques may be used as a diagnostic aid to help improve laboratory performance.

EXAMPLES

Interlaboratory Study 89-6: Metals and Hydrides

Interlaboratory Study 89-6 was conducted in September 1989 to assess the interlaboratory variability for the analysis of metals and hydrides in spiked reagent water. A total of 36 laboratories participated in this study (5).

The traditional Youden two-sample plot was used to assess the results from this study (Figures 1 and 2). To evaluate the results and determine outliers, a pooled standard deviation of the two spiked samples was calculated (5). This acceptance criteria is indicated by the circle on each plot and outlying laboratories are flagged. The location of the outlying results in either the upper right or lower left quadrants, demonstrates either a systemic high or low bias, usually associated with a difference in calibration standards.

Interlaboratory Study 89-5: Acid and Base/Neutral Extractables

Interlaboratory Study 89-5 was conducted in May 1989 to assess the interlaboratory variability for the analysis of acid and base/neutral extractables in spiked reagent water. A total of 15 laboratories participated in this study (6).

The Youden two-sample technique was applied to the results across the GC scan after converting the results to percent recovery of the design value. Tolerance limits of ±20% for performance were assigned (Figures 3 and 4). Interanalyte repeatability can be deemed acceptable if all the data pairs fall within the tolerance limits, as demonstrated in Figure 3. Biases within the scan can be easily recognized as demonstrated in Figure 4.

Interlaboratory Study 89-1: Volatile Organics

Interlaboratory Study 89-1 was conducted in January 1989 to assess the interlaboratory variability for the analysis of volatile organics in spiked reagent water. A total of 16 laboratories participated in this study (7).

To assess the performance across the GC scan, the results were converted to percent recovery of the design value, arranged in approximate order of elution, and plotted as a bar graph (Figure 5). The results demonstrate various patterns of performance. Laboratory 1007 demonstrates a pattern of increasing recovery across the scan, suggesting that the temperature

program may initially be too high, resulting in volatilization of the target parameters. Parameters that are over-recovered are readily identified, suggesting that the calibration standard should be cross-checked for accuracy. The results for Laboratory 1014 indicate a subset of parameters that demonstrate a pattern of decreasing recovery across the scan. This may indicate that the method used by this laboratory is not appropriate for the analysis of these parameters.

CONCLUSION

Graphical presentation of interlaboratory data can facilitate interpretation, and reveal patterns that are not detected by tabular or statistical summaries. They provide a fresh perspective and insights into relationships which may otherwise be overlooked or missed, providing greater incentive to improve laboratory performance.

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FIGURE 1

FIGURE 2 :INTERLABORATORY STUDY 89-6

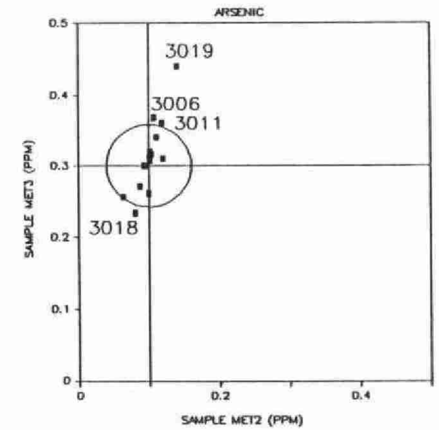

FIGURE 2

FIGURE 12:NTERLABORATORY STUDY 89-6

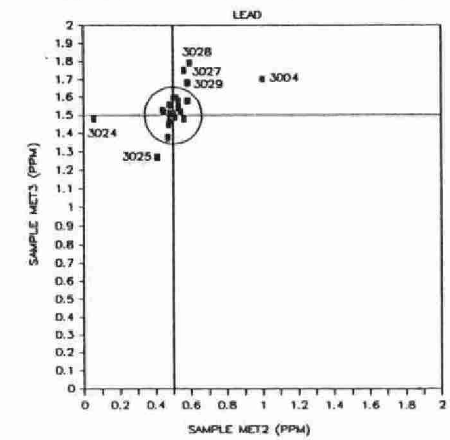

FIGURE 3

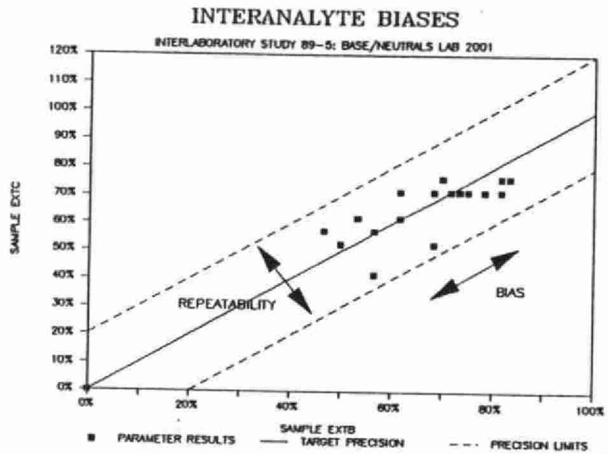

FIGURE 4

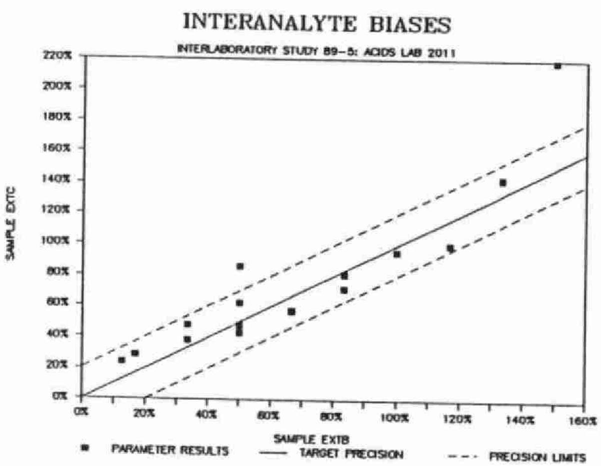

FIGURE 5

FIG. 3 – INTERLABORATORY STUDY 89–1

VOLATILE ORGANICS (MISA GROUPS 16 & 17): SAMPLE VOLB